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Jean Morelle et al.

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Title:

COMPOSITIONS FOR IMPROVING CROP PRODUCTION,

THE QUALITY AND PROTECTION THEREOF

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Commissioner for Patents Washington, D.C. 20231

DECLARATION

Christophe de Mil declares as follows:

- I am a named inventor in the instant application. 1.
- I have worked in the agricultural field for many years and especially in relation to use of amino acids, such as acylated amino acids and their salts, to affect crops. my work has been focussed on both potatoes and grapes. consider myself an agricultural expert with special knowledge of grape and potato physiology.
- 3. I am making this declaration in order to demonstrate the unique properties of copper salts of butyric amino acid on potatoes; the unique properties of copper salts of caprylic amino acid on grapes; and the unique properties of copper salts of

caprylic amino acid on beets. The tests reported herein were performed by me or under my direct supervision and control.

POTATO

- 4. Three different treatments using a copper salt of butyric collagen (C4coCu, sold under the name ECOBIOS) were applied to the potato plants. The treatments differed by the times at which the plants were treated, namely: treatment (a) was administered during the tuberisation phase; treatment (b) was administered at the end of the tuberisation phase (when the potato was 15 to 35 mm in diameter); and treatment (c) was administered when the potato was greater than 35 mm in diameter. For each treatment the dosage was a 30% aqueous solution which was administered at 100 grams per hectare. In each treatment there was a control which was treated with an equal amount of water. The treatment was a foliar spray.
- 5. The details for each treatment (a)-(c) were as follows:

 Treatment (a) Treatment of the potatoes was administered during the tuberisation phase in two tests as follows:
- In France at Bretagne in 1999, a field previously planted with "Delice" potatoes was divided in 2 blocs (each parcel containing 50 plants)
- In France at Somme in 1997, a field previously planted with

"Monalisa" potatoes was divided into 4 blocs (surface of each parcel was $3m \times 0.9m$)

The results from these two tests are reported in Table A attached hereto.

Treatment (b) - Treatment was administered under good weather conditions (T°>18°C), when the potatoes were between 15 and 35 mm, in two tests as follows:

- In France at Somme in 1999, a field previously planted with "Saturna" potatoes was divided in 3 blocs (surface of each parcel was 3m x 0.75m)
- In France at Champagne in 1995, '96, '97 and '98, fields planted with Monalisa" potatoes from 1995 to 1997 and "Russet Burbank" in 1998 were divided in 4 blocs (surface of each parcel was 5m x 0.75m and had the same number of plants).

The results from these two tests are reported in Table B attached hereto.

Treatment (c) - Treatment was administered to the potatoes after the plants had reached a stage of growth of 35 mm in two test as follows:

• In France at Aisne in 2000, a field previously planted with "Monalisa" potatoes was divided in 5 blocs (surface of each

parcel was 18 m²)

• In France in the Ardennes in 2000, a field previously planted with "Saturna" potatoes was divided into 5 blocs (surface of each parcel was 18 m²)

The results from these two tests are reported in Table C attached hereto.

- 6. Another test was conducted to compare the copper salt of butyric collagen (C4coCu, sold under the name ECOBIOS) to the copper salt of caprylic collagen (C8coCu). Identical treatments (doses 100 ml/ha) were administered under good weather conditions (T°>18°C) when the potatoes were at the end of the tuberisation phase (between 15 and 35 mm) as follows:
- In France at Champagne in 1997, a field previously planted with "Monalisa" potatoes was divided in 4 blocs (surface of each parcel was 5m x 0.75m and had the same number of plants)

The results of this test are reported in Table D attached hereto.

7. The results of the experiment of Paragraph 6 were analyzed by a statistical method (Stat ITCF) established according to the works of J.W. Tukey and of H. Scheffe. The analyzation of variance compared the data by groups of 2 parcels and determined coefficient F of Snedecor in each case. Depending

on the degree of freedom in the experiment, the data are significant at 95% or 99%; if this is the case, "significant" is noted in Table D.

- 8. From the test results reported in Tables A-C, it can also be seen that the increased yield for potatoes was only achieved when the potatoes were treated at the end of the tuberisation phase. I find this surprising and unexpected because the prior art DeMil reference (U.S. Patent 4,797,151) makes no mention of treating potatoes at the end of the tuberisation phase to increase yield.
- 9. From the test results reported in Table E, it can be seen that the copper salt of caprylic collagen is less effective than the copper salt of the butyric collagen. I find this to be surprising and unexpected. Furthermore, the prior art DeMil reference makes no mention of this difference.

GRAPES

10. An aqueous solution of copper salt of caprylic collagen (C8coCu) was applied to Pinot Noir grapes at three different stages of growth, namely: at flowering, just after flowering (nouaison) and well after flowering (veraison). Four different plots B1-B4 were tested. Each plot was treated with a 40% aqueous solution at a dosage of 350 grams/hectare (350 ml/ha).

The control received an equal amount of water without the copper salt. Both the weight and the sugar content of the grapes were tested, and the results are reported in Table E attached hereto.

- 11. As can be seen from the results shown in Table E, treatment conducted just after flowering produced the highest yield of sugar while decreasing the weight of the grape. I find this surprising and unexpected, and DeMil makes no mention of increasing the sugar content of grapes using a copper salt of caprylic collagen.
- 12. Tests were run to compare the copper salt of butyric collagen (C4coCu) to the copper salt of caprylic collagen (C8coCu) using the same dosage and treatment method set forth in Paragraph 10 above for both Cabernet Sauvignon grapes and Pinot Noir grapes. Treatment in each case was conducted after flowering. The results of these tests are reported in Table F attached hereto.
- 13. As shown by the results reported in Table F, the copper salt of caprylic collagen was more effective than the copper salt of butyric collagen in increasing sugar content while the reverse occurred in terms of berry weight. I find such results to be surprising and unexpected.

BEETS

- 14. Using a 40% aqueous solution of a copper salt of caprylic collagen, sugar beet plants were sprayed to provide a 150 gram/hectare dose. Three separate treatments were conducted to determine the optimum time for treatment, namely: (1) at the early stages of leafing; (2) during azote accumulation after July 10; and (3) 30 days after the July 10 treatment. In each treatment there was a control group that was given an equal amount of water with the copper salt. The results of these tests are reported in Table G attached hereto.
- 15. As shown in the test results of Table G, it was found that the first treatment increased yield with no effect on sugar content. The second treatment resulted in increased sugar content while the third treatment had no effect on either yield or sugar content. It can also be seen that the first treatment resulted in increased yield of beets.
- 16. The tests of Paragraph 14 were repeated to compare a copper salt of butyric collagen (C4coCu) to a copper salt of caprylic collagen (C8coCu). It was found that the copper salt of butyric collagen increased yield but decreased sugar content of the beets compared to the control while the copper salt of the caprylic collagen increased sugar content of the beets and

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produced a yield compar ble to the control. The results of these tests are reported in Table H attached hereto.

Inteclare further that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Date 12 Just 2002

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Experimental data Potato

a/ Treatment administered during tuberisaton phase

Delice 70.000 plants/ha Bretagne year 1999 variety D location B 50 plants/bloc

		Codibor	Solihers 66 mm	15/5/				900							
			11111 CC	43/33	ПШ	50/4	32/45 mm	28/3	78/35 mm	428 -	<28 mm	_ 2	total	Marketat	//arketable > 45 mm
	Bloc	Weight	Number	Weight	Number	Weight	Number	Weight	Number	Weight	Number	Weight	Number	Weight	Nimber
control group	94	2800	18	13245	131	12340	208	2323	88	767	82	31475	524	16045	149
	B2	2800	32	13100	120	8900	151	1900	69	505	45	30205	417	18900	152
	Average	4300	25	13173	126	10620	180	2112	11	636	8	30840	471	17473	151
Ecobios	B1	1896	11	11400	115	13800	245	2944	114	861	<u>%</u>	30901	563	13296	128
	B2	2367	13	10000	101	11600	208	2205	79	468	52	26640	453	12367	114
	Average	2132	12	10700	108	12700	227	2575	6	665	65	28771	508	12832	120

The Marketable Yield is significantly lower with Ecobios applied during the tuberisation phase and the number of tubercules is higher

40,000 plants/ha year 1997

variety Monalisa 4 location Somme y bloc: 3 m*0,9 m

	Eco	Ecobios	Col	Control
	Weight	Nimber	Weight	Mumbor
Bloc	>40		>40	
B1	6'2	532	11,9	294
B2	6'8	287	13,4	273
B 3	. 9,2	239	12,5	280
B4	9,3	521	13,2	253
Average	8,825	544,75	12,75	275

Experimental data Potato b/ Treatment administered when the potatoes are between 15 and 35 mm in diameter

ECART							
	ECOBIOS	7.56	18.96	32.00	58,52		
moyenne	Control	9,19	18.67	24,00	51,85		Cignificant
		40	40/20	^20	Brut		
	ECOBIOS	6,67	21,33	33,33	61,33	54,67	
bloc3	Control	9,78	16,89	25,33	52,00	42,22	
		<40	40/50	>50	Brut	Total>40 en T/ha	
	Control ECOBIOS	6,67	16,89	31,56	55,11	48,44	
ploc2	Control	8,00	18,22	26,22	52,44	44,44	
		<40	40/50	>50	Brut	Total>40 en T/ha	
	ECOBIOS	6,33	18,67	31,11	59,11	49,78	
ploc1	Control	8,78	20,89	20,44	51,11	41,33	
	T/ha	<40	40/50	>50	Brut	Total>40 en T/ha	
				rcels			
	Satuma	3*3*0,75	irrigué	Homogenous parcels			
Somme				Hom			

+ article : 4 years of significant trials in Champagne by the "Chambre d'Agriculture de Champagne" showing +10 to 20 % of increased caliber yield

Experimental data Potato

c/ Treatment administered after the stage of growth >35 mm

year 2000 location O8300 variety Monalisa Stage 40 mm T°celsius 21°C

97,76 99,37

60,52

27,5

9,74 10,37

Weight < 50 mm | Weight 50-60 mm | Weight > 60 mm | Total Weight

bloc : 18 m²

days after application: T + 89 days 5 repetitions

Non Significant

Ecobios Control

year 2000 location O2270 variety Saturna Stage 40 mm T°celsius 19°C

Control

bloc: 18 m²

days after application: T + 89 days 5 repetitions

	Weight < 30 mm Weight < 45 mm	Weight < 45 mm	Weight > 45 mm Total Weight	Total Weight
B 1	0,92	17,86	65,80	84.58
B2	95'0	16,60	71,18	88,34
B3	0,18	16,06	63,34	79.58
B4	0,22	16,58	67,32	84.12
85	0,26	18,54	96'69	88.76
Average	0,43	17,13	67,52	85.08
81	0,78	18,04	63.94	82.76
B2	99'0	18,44	99'69	88.76
B3	0,34	16,72	66,18	83.24
84	0,42	14,64	72,04	87,10
B5	0,16	18,60	68,94	87.70
Average	0,47	17,29	68.15	85.91

Ecobios

Non significant

Experimental data Potato

Differences in types of lipoaminoacids: Ecobios C4coCu versus C8coCu

Champagne location

year 1997

Monalisa variety

25 mm 20°C Stage T°celsius

bloc: 10 m.

4 repetitions

yield in T/ha

	bloc1	bloc2	bloc3	bloc4	Average	Stat ITCF
Control	504	527	240	515	521,5	8
C8coCu	504	588	283	546	555,25	B A
Ecobios C4coCu	260	603	999	581	575	∢

yield>40 mm in Kg/ha

	bloc1	bloc2	bloc3	bloc4	Average	Stat ITCF
Control	4 843,44	5 064,50	5 189,40	4 949,20	5 011,64	8
C8coCu	4 835,88	5 641,90	5 593,90	5 238,90	5 327,65	B A
Ecobios C4coCu	5 396,16	5 810,50	5 357,60	2 598,50	5 540,69	4

The increased yield with Ecobios is significant and almost 50% better than the results obtained with the C8coCu

location : Beaune year : 1987

variety: Pinot Noir

		Sugar cont	ent (refractome	eter)
	Control group	350 ml /ha during flowering phase	350 ml/ha after flowering (nouaison)	350 ml/ha after the previous phase (veraison)
B1	10,30	10,20	10,80	10,30
B2	10,20	10,30	10,50	10,00
В3	10,10	10,30	10,70	9,80
B4	9,90	10,60	10,60	10,30
Average	10,13	10,35	10,65	10,10
Stat	Α	AB	В	Α

	W	eight of 100 g	rape berries (ii	n grams)
	Control group	350 ml /ha during flowering phase	350 ml/ha after flowering (nouaison)	350 ml/ha after the previous phase (veraison)
B1	191	153	180	193
B2	178	170	195	180
В3	180	167	203	173
B4	186	140	207	184
Average	183,75	157,5	196,25	182,5
Stat	Α	В	AC	Α

location : Beaune year : 1986 Bloc of 15 plants Test performed on 200 berries

variety: Cabernet Sauvignon

	Sugar co	Sugar content (refractometer)	tometer)
	Control group	C4ccU	റയാ
B1	02'6	9,40	06'6
B2	06'6	06'6	10,30
В3	09'6	09'6	10,20
Average	9,73	9,40	10,13
Stat	٧	8	၁

	weight of 10	weight of 100 grape berries (in grams)	es (in grams)
	Control group	C4∞CU	C8coCU
81	125,00	153,00	130,00
B2	115,00	149,00	127,00
B3	136,00	147,00	135,00
Average	125,33	149,67	130,67
Stat	4	В	∢
			1

variety: Pinot Noir

	Sugar co	Sugar content (refractometer)	tometer)
	Control group	C4coCU	വാ∞ജാ
B1	10,20	08'6	10,45
B2	10,10	02'6	10,60
ВЗ	08'6	09'6	10,50
Average	10,03	29'6	10,52
Stat	٧	8	3

significant C4coCu decrease sugar content and increase yield C8coCu increase quality with higher sugar content

			ı		Γ	_
ss (in grams)	CBcoCU	182,00	190,00	180,00	184,00	٧
weight of the grape perries (in grams)	C4∞CU	196,00	215,00	192,00	201,00	В
WEIGHT OF TO	Control group	170,00	185,00	179,00	178,00	A
		B1	B2	83	Average	Stat

Sugar Beets location : Meharicourt (80) year : 1989 4 Blocs

15 plants for ach modality variety : Ariska

	Sugar content						
	Control group	150 ml /ha during early 150 ml/ha during azd stage of leafing accumulation at july		150 ml/ha after the previous phase july 10 + 30 days			
B1	15,8	15,9	17,2	16,2			
B2	17,1	16,5	18,0	16,8			
В3	16,8	16,7	17,9	17,0			
B4	17.5	17,0	18,8	17.7			
Average	16,8	16,5	18,0	16,9			
Stat			significant				

Γ	Yield in Kg							
	Control group	150 ml /ha during early stage of leafing accumulation at july 10		I previous prace this it +				
B1	17,6	18,7	17,4	17,2				
B2	16.8	17,3	17,2	17,0				
B3	17,3	18,3	17,8	17,5				
B4	16,4	17,5	17,0	16,7				
Average	17,0	18,0	17,4	17,1				
Stat		significant						

Sugar Beets location : Vigny (95) year : 1990 4 Bloc 12 plants for each modality variety : Accord

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Sugar content	വാമ്മാ	18,2	19,2	18,4	18,7	18,6	၁	
	C4coCU	16,2	17,4	16,7	16,2	16,6	8	significant
	Control group	17,3	18,2	17,5	17,4	17,6	А	
		B1	82	E8	B4	Average	Stat	

		_		_				
	റാംജാ	37,8	29,9	2'98	29,7	33,5	Y	
Yield in Kg	C4coCU	38,6	35,1	38,3	34,2	9'98	В	significant
	Control group	33,7	28,7	32,3	30,7	31,4	٧	
		B1	B2	B3	B4	Average	Stat	